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Fine China

Trials with Ontario Minerals

by
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Dennis Salt Consultants

edited by
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METRIC CONVERSION FACTORS

Conversion from SI to Imperial			Conversion from Imperial to SI		
SI Unit	*Multiplied by*	*Gives*	*Imperial Unit*	*Multiplied by*	*Gives*
		LENGTH			
1 mm	0.039 37	inches	1 inch	**25.4**	mm
1 cm	0.393 70	inches	1 inch	**2.54**	cm
1 m	3.280 84	feet	1 foot	**0.304 8**	m
1 m	0.049 709 7	chains	1 chain	20.116 8	m
1 km	0.621 371	miles (statute)	1 mile (statute)	**1.609 344**	km
		AREA			
1 cm^2	0.155 0	square inches	1 square inch	**6.451 6**	cm^2
1 m^2	10.763 9	square feet	1 square foot	**0.092 903 04**	m^2
1 km^2	0.386 10	square miles	1 square mile	2.589 988	km^2
1 ha	2.471 054	acres	1 acre	0.404 685 6	ha
		VOLUME			
1 cm^3	0.061 02	cubic inches	1 cubic inch	**16.387 064**	cm^3
1 m^3	35.314 7	cubic feet	1 cubic foot	0.028 316 85	m^3
1 m^3	1.308 0	cubic yards	1 cubic yard	0.764 555	m^3
		CAPACITY			
1 L	1.759 755	pints	1 pint	0.568 261	L
1 L	0.879 877	quarts	1 quart	1.136 522	L
1 L	0.219 969	gallons	1 gallon	**4.546 090**	L
		MASS			
1 g	0.035 273 96	ounces (avdp)	1 ounce (avdp)	28.349 523	g
1 g	0.032 150 75	ounces (troy)	1 ounce (troy)	**31.103 476 8**	g
1 kg	2.204 62	pounds (avdp)	1 pound (avdp)	**0.453 592 37**	kg
1 kg	0.001 102 3	tons (short)	1 ton (short)	**907.184 74**	kg
1 t	1.102 311	tons (short)	1 ton (short)	**0.907 184 74**	t
1 kg	0.000 984 21	tons (long)	1 ton (long)	**1016.046 908 8**	kg
1 t	0.984 206 5	tons (long)	1 ton (long)	**1.016 046 908 8**	t

Note: Conversion factors which are in bold type are exact. The conversion factors have been taken from or have been derived from factors given in the Metric Practice Guide for the Canadian Mining and Metallurgical Industries, published by the Mining Association of Canada in cooperation with the Coal Association of Canada.

Fine China

Trials with Ontario Minerals

Summary

A preliminary assessment of the feasibility of using provincial minerals in the manufacture of fine china of premier quality was commissioned by the Ontario Ministry of Northern Development and Mines in July 1986. Trial products were fabricated and evaluated with respect to the composition and standards of commercial bone china.

The trial replacements for bone ash, china clay and flux materials used in bone china were beneficiated samples of rock phosphate and kaolin clay from the Cargill Township and the Mattagami River basin areas respectively in northern Ontario, as well as commercial ceramic-grade nepheline syenite and silica products from the province.

Although the trial products with substitute materials did not achieve the translucency and whiteness of bone china, the tests indicated that the Ontario minerals were suitable for the manufacture of two distinct grades of china, namely:

a. a fine translucent china with an attractive ivory colour, judged to be suitable for tableware and giftware of high quality. Acceptable translucency however, required that the rock phosphate be kept below about 20% by weight of the ceramic body.

b. a pale brown, opaque but sound body with higher phosphate content. This body with opaque glazing and lithographic or other decoration was considered to be a competitive prospect in the modestly-priced range of tableware and giftware.

Ceramic-grade nepheline syenite and silica products performed well without further treatment; however, both the rock phosphate and kaolin clay from northern Ontario would require substantial upgrading to meet commercial standards for the manufacture of fine china tableware.

1. Introduction

The fabrication and evaluation of fine china bodies using materials from Ontario was commissioned by the Mineral Development and Lands Branch of the Ministry of Northern Development and Mines in 1986. The services of Dennis Salt Consultants of Pefferlaw, Ontario, were contracted in July of that year to conduct the study. Funding was provided by the Economic Development Program of the Canada–Ontario Mineral Development Agreement (COMDA), which came into effect on April 1, 1985. COMDA is a subsidiary agreement to the Economic and Regional Development Agreement (ERDA) signed by the Governments of Ontario and Canada in 1984.

The principal aim of the project was to explore the feasibility of producing a porcelain body, of equivalent quality to commercial bone china, using provincial mineral resources. A typical body composition for bone china, expressed as a percentage of total solids, is 50% bone ash, 25% china clay and 25% flux (Cornish stone, a mixture of feldspar, silica and kaolin). Ontario is an established producer of porcelain–grade silica and feldspathic material (nepheline syenite). The province also has promising resources of kaolin and rock phosphate in northern Ontario. Comparisons were to be made between the trial raw materials and traditional English ceramic materials in standard body composition for bone china.

The body trials were arranged in two consecutive stages. The first stage was the evaluation of Ontario rock phosphate as a suitable replacement for animal bone ash. The second stage was the evaluation of Ontario silica, kaolin and nepheline syenite, together with phosphates, in replacement of imported kaolin and flux materials. As the study was a preliminary assessment, no extraordinary measures were taken with respect to the processing and purity of Ontario kaolin and phosphate materials beyond comminution and reduction of the level of iron–bearing contaminants.

Canadian imports of bone china products were valued at $36.2 million in 1986 (Statistics Canada, verbal communication). There was only one small producer of bone china in Canada at the time of the study; this producer in Toronto, Ontario, used imported raw materials in the manufacture of its products.

DEFINITIONS

Porcelain is a ceramic product of dense, white, translucent character. The body of standard porcelain consists of a mixture of kaolin clay, feldspar and silica.

Porcelain is often differentiated (Schüller 1979) according to the firing temperatures. Porcelain fired at temperatures between 1380°C and 1460°C is called "hard porcelain", whereas "soft porcelain" is fired at temperatures as low as 1200°C. Porcelains are sometimes also classified by their application, such as sanitary porcelain, electro–porcelain, tableware porcelain and chemical porcelain.

Bone china is a type of porcelain. It is produced mainly in England and the main constituent is bone ash. A standard British definition (Dinsdale 1967) refers to a minimum 30% content of phosphate derived from animal bone and calculated as tricalcium phosphate. The fired product is composed (St. Pierre 1954) of tricalcium phosphate, anorthite and a glassy phase; it exhibits a microstructure that differs considerably from other types of porcelain.

Although the terms *porcelain* and *china* are often used interchangeably, porcelain is the preferred term in Europe. In Britain, the term *china* is confined to bone china. In the United States, the terms are applied on the basis of use — porcelain usually refers to types with technical applications, whereas china is used broadly for types with non–technical applications, regardless of whether bone ash is or is not part of the composition of the product.

Fine china is defined, for the purposes of this study, as a type of true porcelain used in tableware and artware and which features the qualities, notably superior translucency, associated with bone china.

2. History

Porcelain or china was developed by the Chinese as early as the 12th century. Porcelain bodies were formed from a mixture of 'petuntse' or china stone (a feldspathic rock) and kaolin (white china clay). European interest in Oriental porcelain was stimulated by Marco Polo in 1271; journals of his 3–year expedition into China extolled the virtues of the beautiful 'porcellana' produced by the Chinese potters. Once the trade routes were opened in the 14th and 15th centuries, the great porcelains of China were brought to the eager hands of European gentry and nobility.

Soon the race was on to be the first in Europe to reproduce Chinese translucent porcelain. Kings and other rulers subsidized factories so that their country would hold the honour of this new discovery. It was at the Meissen factory in Germany that the first translucent porcelain was made at the turn of the 18th century.

China, utilizing bone ash, was first introduced in 1794 by Josiah Spode II in England. He used calcined ox bones to produce a very white, translucent body.

Ceramists have at various times investigated the possibilities of substituting rock phosphates or synthetic calcium phosphates for bone ash. In 1901, pumice was tried in place of bone, based on the assumption that similar physical structure would result in comparable translucency, but such attempts were not successful (Moss 1902). Trials with apatite (Davis 1919) and chemically prepared tricalcium phosphate (German 1950), reported deformation of the china body. Most of the experiments reported important shortcomings in the trial china bodies, which have been summarized (Taylor *et al.* 1979) as follows:

- poor fired colour with untreated rock phosphates, notably buff and brown colours when iron is present, and green colours if the chromium content of the rock phosphate exceeds 50 ppm. Another significant impurity in rock phosphate is fluorine, which can be removed by defluorination or chemical treatment. The latter type of processing produces dicalcium and tricalcium phosphate;
- high firing contractions of bodies with synthetic calcium phosphates;
- lower vitrification temperatures and shorter firing ranges for bodies containing dicalcium phosphate;
- cracking when drying some types of moulded ware.

By the late 1960's, chemically–prepared phosphates, hydroxy–apatite and anhydrous dicalcium phosphate were successful substitutes in achieving the desired translucency (Ashworth *et al.* 1969). By the early 1970's, bleached phosphate rock (low in chromium and iron), was substituted with apparent success for bone ash (Albright & Wilson Ltd. 1971). A satisfactory replacement was also reported (Taylor *et al.* 1979) by a calcined and ground mixture of dicalcium phosphate and limestone to produce china with an appearance and properties closely resembling those of bone china.

Previous work investigating the substitution of Ontario kaolin for English china clay in fine china bodies was undertaken in December of 1985 by Dennis Salt using kaolin from the Mattagami River deposit in northeastern Ontario. After the removal of silica and iron from the kaolin, trial bone china plates were successfully produced at the Sunrise Fine Bone China Company in Toronto. The bone china plates were vitreous and of good translucency. When held to the light, the pieces exhibited a faint ivory colour, a property judged to be aesthetically pleasing and acceptable in the market.

3. Materials

TRADITIONAL BONE CHINA MATERIALS

Bone ash, Cornish stone and china clay are the classical ingredients in a bone china body. The low plasticity and narrow firing range of the body, however, require good quality control of the raw materials as well as excellent process control through all stages of manufacture. The typical specifications of these materials used as standards in the present study are outlined in Table 1 on page 10.

Bone Ash

Bone ash is calcined bone and is composed almost entirely of hydroxy-apatite, $Ca_{10}(PO_4)_6(OH)_2$. Cattle bones, virtually iron-free, have always been the preferred source of supply.

Until the second decade of this century, England's bone supplies came from the Rio Grande and River Plate areas of South America. Supplies for a time came from England itself, but with the decline in the glue industry, in the processing of bone, and partly due also to the British preference for buying its meat on the bone, raw bones are now mostly obtained from the Benelux countries of Europe. The final processing of the bone is done in England.

Bone is composed of minute crystals of calcium phosphate in a matrix of the fibrous protein collagen. Collagen, the precursor of gelatine, is removed during processing. Partial removal of organic matter is achieved by water or solvent extraction (degreasing and de-glueing). The calcium phosphate residue, referred to as de-gelatinized bone in the tableware industry, has the same crystal structure as the rock phosphate hydroxy-apatite, the chemical analysis of which corresponds closely to that of calcined bone (Webster *et al.* 1987).

The bone is next passed through a calcining process, during which the crushed and de-gelatinized bones are heated to remove residual organic matter and to produce, with increasing temperature, sintering and crystal growth. The resultant material has increased bulk density and hardness. The bone ash is then water-ground to the desired fineness (generally 80% by weight under 10 microns), and allowed to age or "ark" for a period of six or more weeks, allowing further chemical changes and enhancing the workability of the body. Finally, the calcined bone is dried to 10–15% moisture content to remove air from the ground bone ash slip (Rado 1981).

Cornish Stone

Cornish (or China) stone is the principal flux material, lowering the fusion point of the ceramic batch with the introduction of alkali feldspar minerals – potassium feldspars $K[AlSi_3O_8]$ and sodium feldspars $Na[AlSi_3O_8]$.

Cornish stone is a type of altered granite rock that is the chief source of feldspar in Britain. It contains, in addition to feldspar, quartz (silica) and mica, small amounts of kaolin and traces of fluorine-bearing minerals. The alteration of the granite has resulted in varying degrees of decomposition of the feldspars to kaolinite and there are consequently varieties ranging from "hard purple" to "soft white". Blending of the varieties is practised in order to provide a suitable range of fusion in china bodies (Worrall 1982).

The mixed stone is water-ground (60% by weight below 10 microns), screened and passed through a magnetic separator. It is then washed, filtered and dried to 10–15% moisture content.

There is a certain amount of inconsistency in the composition of Cornish stone, nevertheless, and it has been replaced in some bone china bodies by other alkali feldspar sources and a small amount of quartz (Rado 1981).

China Clay

China clay or kaolin are terms used interchangeably for a white-firing clay with kaolinite, $Al_2[Si_2O_5(OH)_4]$, as the dominant mineral. It occurs either as residual (primary) deposits formed by the in-situ alteration of feldspar-bearing igneous rocks (e.g. Cornwall, England) or as sedimentary (secondary) re-deposited kaolin clays such as those of Georgia, USA.

The English bone china industry has relied almost exclusively on Cornish china clay for its plastic material. This residual clay also contains quartz, feldspar and mica minerals which remain from the parent rock. Hydraulic mining in the pits produces a clay-water suspension which is pumped into tanks, where some quartz and mica are removed by sedimentation. After removal of excess water by settling over a long period or by filter-pressing, the clay is dried in kilns. The china clays are blended into various grades according to criteria such as particle size and wet-to-dry contraction. Cornish china clay is one of the purest sources of kaolinite, forming 75–85% of the material. The blended clay (grolleg) as used in this study has an approximate mineralogical composition (by weight) of 78% kaolinite, 17% "micaceous" material and 4% quartz (English China Clays Group, England).

ONTARIO TEST MATERIALS

Ontario has significant deposits of both proven and potential ceramic raw materials to replace the classical ingredients in china manufacture. The province is the Western World's leading producer of feldspar-rich nepheline syenite and is also a major producer of ceramic-grade silica; a combination of these materials was selected as the test replacement for Cornish stone. The potential substitute for English china clay is kaolin clay from extensive deposits in northeastern Ontario. The same region also hosts several large apatite-bearing deposits — the rock phosphates — to provide a possible alternative to calcined bone. The source locations of the alternative materials used in the body trials are shown in Figure 1 and their specifications are presented in Table 2 on page 11.

Ontario Phosphate

Several phosphate deposits associated with carbonatite intrusive rocks are known in northern Ontario, including the Cargill Township, Martison Lake and Lackner Lake occurrences. Geological and metallurgical work has indicated the Cargill deposit may have the highest potential for initial development, and samples for this study were obtained from this location.

The Cargill deposit, located 32 km southwest of Kapuskasing, features fresh and leached carbonatite blanketed by a weathered residuum rich in apatite, $Ca_5(PO_4)_3(OH,F,Cl)$. Apatite content, estimated at 5–15% in the fresh carbonatite, is at levels of 20–98% in the residuum. The residuum, an unconsolidated sand, also contains varying quantities of rare earths, vermiculite, niobium-bearing minerals, clay minerals and quartz sand (Sandvik and Erdosh 1977).

Samples were taken from a phosphate concentrate stockpiled at the deposit; this material from the residuum had been beneficiated by crushing, grinding, attrition scrubbing and desliming.

The first sample batch (Batch 1) was submitted to the Ontario Research Foundation (ORF) in Mississauga to obtain reductions in particle size (minimum 80% minus 10 microns) and in iron content (to below 1% Fe_2O_3). The batch, after primary grinding, was passed through a scrubber (70% solids content, retention time of 5 minutes) and the resultant slurry was deslimed. The plus 325 mesh fraction was subsequently ground for 1.5 hours in a pebble mill and passed twice through a high intensity magnetic separator. Fine grinding in a porcelain ball mill of sample portions for 4, 5 and 6 hours gave products ranging in particle size from 80% minus 10 microns (4-hour grind) to 95% minus 10 microns (5- and 6-hour grinds). Fe_2O_3 content, however, was only lowered from 1.83% (ORF head sample) to 1.45% (product).

A second batch (Batch 2) was treated by ORF to obtain a more finely-ground product for comparative body trials. The same comminution procedures as for Batch 1 were followed to provide an 83% minus 5 microns product. Batch 2 was, however, subjected to four passes in new magnetic separation in a greater effort to lower the iron content - one low-intensity pass and three high-intensity passes with a more dilute solids content to ensure good separation. This resulted in a substantial lowering of the iron content in the Batch 2 product to 0.45% Fe_2O_3.

Apatite with minor calcite, monazite and hematite were identified at ORF by X-ray diffraction analysis as the principal minerals in Batch 2.

Although it is recognised that some of the minor elements in the Cargill phosphate, notably fluorine, may require that the material be further treated to meet acceptable levels for ceramic use, no additional processing was undertaken for this preliminary study of the potential use the sampled phosphate.

Nepheline Syenite

Nepheline syenite is a quartz-deficient igneous rock composed mainly of alkali feldspars, $(K,Na)[AlSi_3O_8]$ - albite and microcline - and the feldspathoid mineral nepheline, $Na_3(Na,K)[Al_4Si_4O_{16}]$. Varying amounts of accessory minerals in the rock include mica, hornblende and magnetite.

Nepheline syenite has been mined since the 1930's at the Blue Mountain deposit located near Nephton in Methuen Township. Mining is open-pit, and the feed to the plant is crushed in two stages to minus 5 cm. After drying, the material is crushed further and then ground to minus 30 mesh size. Magnetite is removed in low-intensity magnetic separators. Following additional screening and classification, the nepheline syenite sand is passed through high-intensity magnetic separators for the removal of garnet, biotite mica and hornblende and constitutes glass-grade material. Ceramic grades require further grinding in pebble mills together with air classification.

The product grade selected for the trials showed a particle sizing not exceeding 30 microns, averaging 7.5 microns. The material is valued as a ceramic flux, notably for its high alkali and low iron content as well as for the consistency of its composition.

Silica

A high-grade Precambrian quartzite rock (98.5% SiO_2) has been quarried since 1970 on Badgeley Island in Georgian Bay, 6 km west of Killarney in northern Ontario.

Mining is conducted between May and November; pit-run material is processed in a two-stage crushing and wet screening plant on Badgeley Island. Minus 2-inch material is shipped over 225 km to Midland on southern Georgian Bay. Dry milling is carried out at Midland. It consists of drying, crushing and ball milling; subsequent screening and classification produces sand-sized grades for the glass industry. Pebble milling of undersize

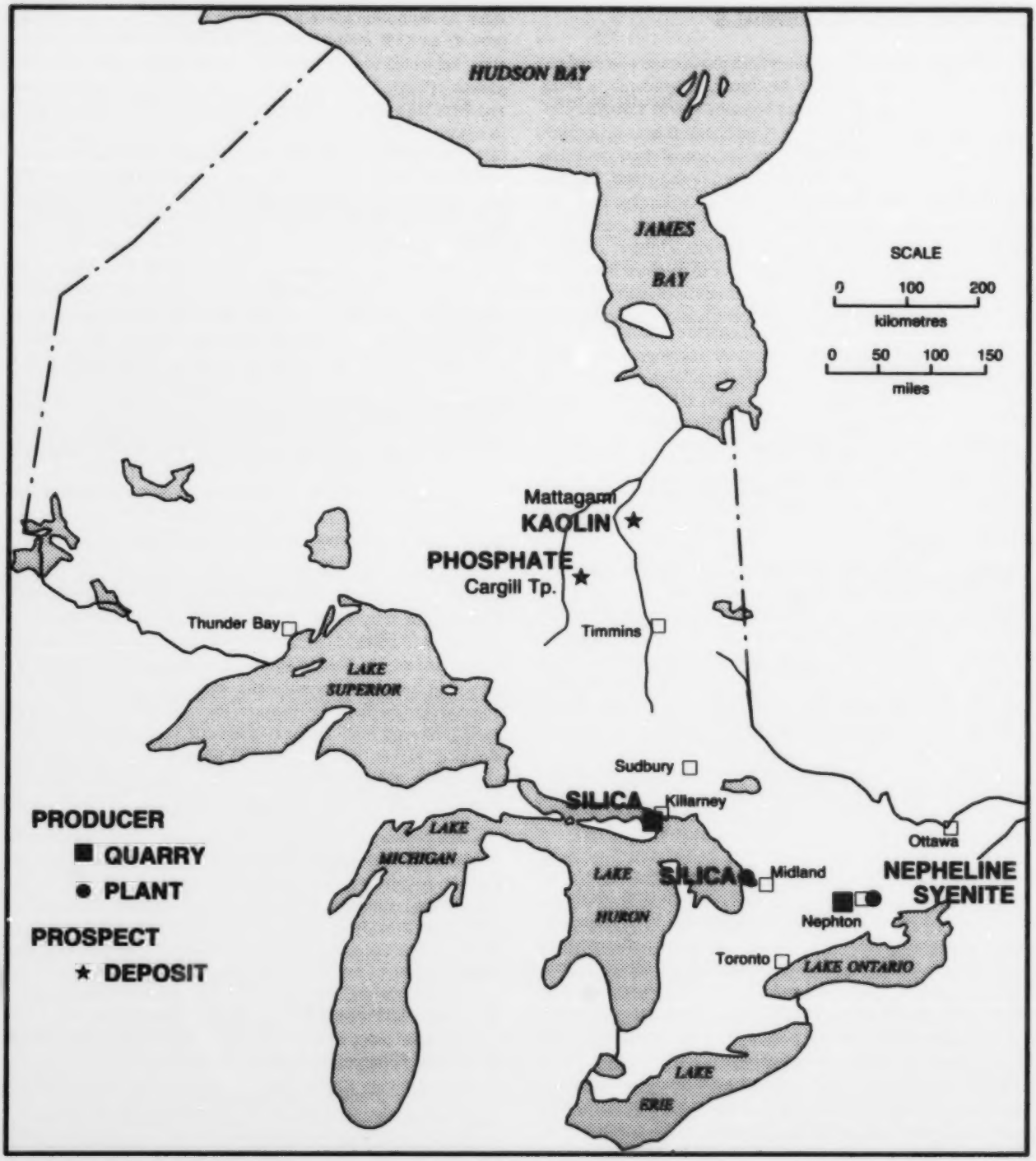


FIGURE 1. LOCATION MAP SHOWING SOURCES OF TEST MATERIALS FOR FINE CHINA.

material provides the ceramic-grade silica product of minus 325 mesh (40 microns) used in this study.

Kaolin Clay

Kaolinitic clays associated with quartz sands underlie an area of approximately 100 km by 50 km in the Moose River Basin, southwest of James Bay in northern Ontario. This sedimentary deposit, frequently over 100 m in thickness, is overlain by thick marine sediments and glacial tills.

Kaolinite is the major clay mineral; it is generally well-crystallised and is particularly dominant in the minus 2 microns fraction. Minor amounts of quartz, chlorite and illite are usually present (Vos 1982).

The kaolin sample used in the study was taken from the eastern bank of the deeply-incised Mattagami River, near Smoky Falls in Kipling Township. After the preliminary removal of some quartz by washing, the coloured sample was submitted to I.M.D. Laboratories Ltd. for bleaching treatment to improve the wet and dry brightness. The best results were obtained by using an "oxidizing" treatment at 60°C followed by a reducing treatment at a pH of 2.5 and at the same temperature. The bleached material was then filtered to remove the bleaching solution containing the solubilized components. The filter cake was re-slurried, allowed to settle and re-filtered. Both the chemical analysis of the bleached re-filtered kaolin and X-ray diffraction indicated the presence of a significant amount, possibly as high as 10%, of free quartz in the sample.

TABLE 1. TRADITIONAL BONE CHINA MATERIALS – SPECIFICATIONS.

MATERIAL	CHEMICAL COMPOSITION		PARTICLE SIZE DISTRIBUTION
BONE ASH	**Major elements (wt %)**		**80% minus 10 microns**
	CaO	53.90	
	P_2O_5	41.90	
	SiO_2	0.34	
	Al_2O_3	0.05	
	Fe_2O_3	0.05	
	MgO	0.91	
	K_2O	0.02	
	Na_2O	1.38	
	TiO_2	0.01	
	LOI	1.19	
CORNISH STONE (China Stone)	**Major elements (wt %)**		**60% minus 10 microns**
	SiO_2	72.38	
	Al_2O_3	14.83	
	Fe_2O_3	0.29	
	MgO	0.11	
	CaO	1.77	
	K_2O	4.60	
	Na_2O	3.29	
	TiO_2	0.02	
	LOI	1.87	
CHINA CLAY (Grolleg)	**Major elements (wt %)**		**Typical: 87% minus 10 microns 47% minus 2 microns**
	SiO_2	47.70	
	Al_2O_3	37.20	
	Fe_2O_3	0.60	
	MgO	0.25	
	CaO	0.10	
	K_2O	1.84	
	Na_2O	0.08	
	TiO_2	0.03	
	LOI	12.06	

Sources for analyses: *Bone ash and Cornish stone*, Jesse Shirley and Son Limited, England; *China clay*, English China Clays Group, Anglo-American Clays Corporation, England; Worrall (1982).

TABLE 2. ONTARIO TEST MATERIALS — SPECIFICATIONS.

ROCK PHOSPHATE
(Partially Beneficiated)

CHEMICAL COMPOSITION

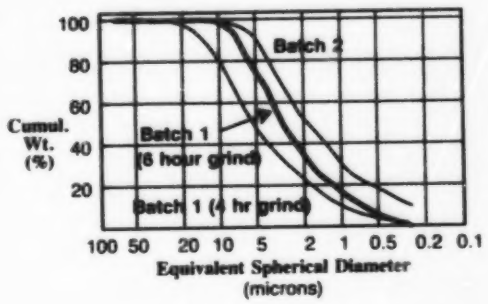
Major elements (wt %)

	Batch 1	Batch 2
CaO	49.4	50.8
P_2O_5	38.3	39.9
SiO_2	6.41	5.39
Al_2O_3	1.36	1.01
Fe_2O_3	1.45	0.45
MgO	0.44	0.32
K_2O	0.16	0.13
Na_2O	0.38	0.14
TiO_2	0.26	0.10
MnO	0.04	0.02
F	2.31	—
LOI	1.85	1.77

Trace elements (ppm)

Cr	< 10	170
Rb	10	10
Sr	2970	3140
Y	240	240
Zr	640	
Nb	50	30
Ba	140	110
Cl	350	
U	23.0	

PARTICLE SIZE DISTRIBUTION



NEPHELINE SYENITE
(Beneficiated Product)

Major elements (wt %)

SiO_2	60.84
Al_2O_3	22.9
Fe_2O_3	0.10
MgO	0.08
CaO	0.57
K_2O	4.83
Na_2O	9.54
LOI	1.14

microns	cumul. wt %
-1	7
-2	15
-3	22
-5	34
-8	56
-10	68
-15	88
-20	96
-30	100

SILICA
(Beneficiated Product)

Major elements (wt %)

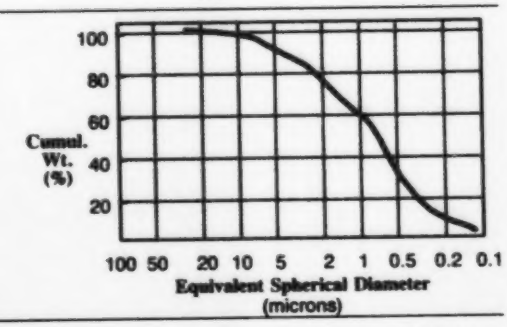
SiO_2	98.97
Al_2O_3	0.56
Fe_2O_3	0.07
MgO	0.02
CaO	0.02
K_2O	0.15
Na_2O	0.01
TiO_2	0.06
LOI	0.15

microns	cumul. wt %
-1	5
-2	10
-3	14
-5	24
-8	37
-10	46
-20	77
-30	94
-40	99

KAOLIN CLAY
(Partially Beneficiated)

Major elements (wt %)

SiO_2	51.0
Al_2O_3	32.7
Fe_2O_3	0.61
MgO	0.46
CaO	0.41
K_2O	0.38
Na_2O	0.08
TiO_2	0.58
LOI	13.8



Sources for analyses: *Rock phosphate*, Ontario Research Foundation, Mississauga, Ontario; *Nepheline syenite and Silica*, Technical Centre, Indusmin Division of Falconbridge Ltd., Toronto, Ontario; *Kaolin clay*, I.M.D. Laboratories Ltd., Don Mills, Ontario.

4. Methodology

BODY PREPARATION

Slip Formulation

The slip casting method was used in all body trials. It was essential that the total solids and water content should be standardized. Brogniart's Formula was used to calculate the amount of dry material and water to be used with each formulation. Brogniart's Formula is expressed as follows:

$$D = (P-20) \times [SG \div (SG - 1)]$$

where

D = dry content of slip
P = pint weight of slip
SG = specific gravity of materials in slip

A total capacity of just over 3 gallons of slip was chosen. Since the ideal casting slip, for maximum pourability, minimum shrinkage and optimum draining qualities should be at a density of 1.7875 or 35.75 ounces per pint, the dry content of the slip was calculated as follows:

$$\text{Dry content} = (35.75 - 20) \times (2.5 \div 1.5)$$
$$= 26.25 \text{ ounces per pint of slip}$$

Dry content of slip for one gallon at 26.25 ounces per pint is equivalent to 13.125 pounds. The weight of water per gallon of slip was calculated as follows:

$$\text{Water content} = (35.75 - 26.25) \times (8 \div 16)$$
$$= 4.75 \text{ lbs/gallon}$$

Body Stain

Normal bone china practice is to add approximately 0.0125% by weight of cobalt chloride, based on the total dry content of the mix. This has the effect of developing a completely white body. This proportion was used in body mixes, unless otherwise noted in the separate body trial reports.

PHOTO 1. *Pouring of clay slip into mould as it is carefully "whirlered" to prevent pin-holing and wreathing marks.*

Slip Deflocculation

Standard manufacturing practice indicates that the ideal fluidity for a casting slip is obtained when the slip registers between 240 and 260° on a Gallenkampf torsion viscometer (using a 30 gauge wire and an 11/16 inch stainless steel bob). All bodies were deflocculated to this level, using sodium silicate with a specific gravity of 1.375. This corresponded to 75° on the Twaddle scale, calculated as follows:

Degrees Twaddle = (S.G. - 1) x 200

The procedure was to first correct the pint weight or specific gravity of the slip, then incrementally add the sodium silicate until the required fluidity was reached.

Mixing Procedures

Cobalt chloride stain, which had previously been measured and then dissolved in boiling water, was added to the measured volume of water. Kaolinite and Cornish stone (or during mixing trials, nepheline syenite, and in later trials silica) were added in sequence. Half of the sodium silicate was added to the mix, followed by the phosphate and then by the second half of the sodium silicate. The slip was mixed for 2 hours at high speed, during which the pint weight (or density of slip) and the fluidity were checked and corrected by adding refining amounts of deflocculant as required. The slip was passed after mixing through a 150 mesh sieve and run over a high-intensity magnet.

SLIP CASTING

A working model was needed prior to casting the china pieces. Sculptures of the required shape were made from clay, plaster or wax. A master or block mould was made using plaster of paris from the original sculpture. A subsequent "positive" form (the case-mould) was cast from the master mould in plaster, reproducing the original sculpture. Plaster was then poured into the case-mould, forming another "negative" mould; it was this working mould into which the slip was poured (Photo 1) and a cast was made (Photo 2).

Water immediately passed from the slip into the porous mould, leaving behind a frail skin of clay shaped by the mould. The longer the slip was left in the mould, the thicker was the formation of the clay skin (Photo 3). After standing for 2-3 minutes, the clay formed a shell and, once the piece had achieved its pre-designated thickness, the slip remaining inside the newly-formed clay piece was carefully poured out. This effluent was passed through a 150 mesh sieve and was available for re-use in casting the next piece.

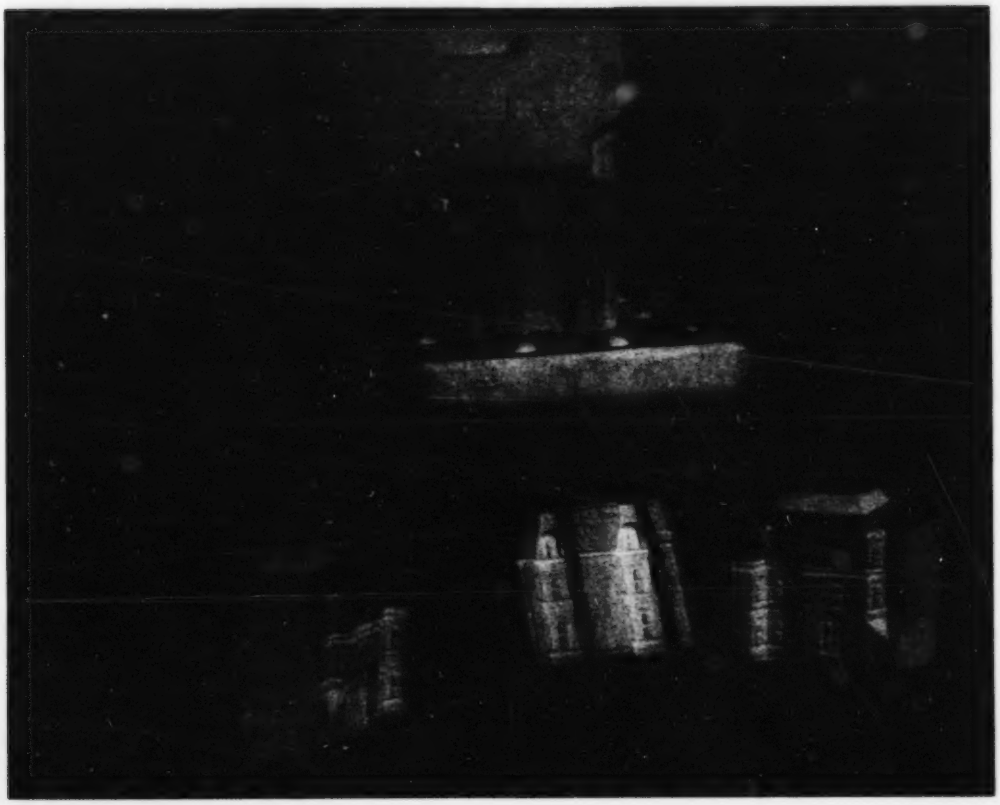


PHOTO 2. *A clay cast, shown with the five parts of a plaster of paris mould.*

PHOTO 3. *Bowl mould filled with slip. Water from the slip passes into the mould, causing a shell of clay to be formed on the interior surface.*

In the pouring process, care was taken not to introduce the slip into the mould too quickly, since air bubbles would be entrapped or swirl-marks developed in the cast piece. When filling by hand, as with small-quantity trials, a gentle whirling of the piece on a standard 8-inch bench whirler ensured the even introduction of the slip and its gentle distribution around the mould, so as to eliminate pinholes and casting marks.

Varying with the size of the piece, a normal bone china body will cast up in a 3–5 minute period. All of the bodies detailed in this report were in the same range of casting-up times.

When tipping the surplus slip from the mould, care was taken to tip out the slip in one unimpeded motion, passing the mould through a 121° motion from the horizontal within a 2-second period. The mould was carefully held at the 31° angle of inversion until the slip drippings ceased. The cast, now going through its final dripping/draining stage, was placed onto a draining stand set at the ideal 31° angle. The correct angle is important, in that no back-draining can take place which would leave either permanent marks on the inside walls of the piece or stalactitic forms hanging from the interior of the inverted piece.

After draining for approximately 5 minutes, the mould was taken off the draining rack. The clay over-fill (left on the top of the mould deliberately in the casting process to allow for the drop in slip-top level as water in the slip passes into the mould below) was carefully scraped off, using a tungsten-carbide knife as the piece was again whirled to ensure a smooth action.

The piece was then free to contract in the mould without cracking. After half an hour (or longer with pieces of complicated shape), the piece was removed from the mould and left to dry. It was then ready for sponging and fettling.

Once the clay piece was completely dry, mould seam-marks were gently stroked away, using a small tungsten-carbide knife. Using a fine sponge, or smooth-hair pencilling brush, any blemishes on the clay piece were carefully smoothed (Photo 4). Once the water used in the sponging operation had dried from the surface, the piece was ready for firing.

FIRING

In the first or 'biscuit' firing, bone china in its initial clay state is normally fired at a temperature of approximately 1250°C, with a 'soaking' period (during which the kiln is held at the peak temperature) of 30–60 minutes.

A combination of factors necessitates the use of refractory supports for most pieces. Such factors include body composition, the actual firing temperature at which the piece goes ductile, and the speed of vitrification (the period during which pore-spaces are closed). The body's final 20% of porosity is lost in the upper 30° of firing. In the case of cups, the pieces were placed inverted on to a dome-shaped or slanting-angle ring, whilst plates and other flatware items sat in custom-made formers or setters which supported the piece in its final fired and contracted position. Bone china, like most vitreous bodies, contracts about 12.5% from modelled to fired size. The bodies developed in these trials had the same overall contraction.

With virtually all of the pieces produced in the course of this work, refractory rings were used as is the usual practice for bone china (Photo 5). It was only in the last stage of production, when a bowl of 8.5 inch diameter was placed (and for which there was no refractory ring), that an experiment was made in firing the piece in a free-standing upright position. The bodies survived the biscuit firing without distortion. This would not have been possible with a bone china piece of this size and shape.

Most of the developed bodies were fired at temperatures considerably lower than that for bone china. The translucent body was fired at 70°C lower than bone china, which could translate into considerable savings in fuel and firing time.

GLAZING

After firing, pieces were dipped in a normal transparent low-solubility glaze (Photo 6), such as is conventionally used in the bone china industry. The ingredients of the glaze had been pre-fired with silica so as to render the glaze non-toxic both in factory use and later by the consumer.

The glaze in the dipping process was carefully controlled as to density (1.725) or pint weight (34.5 ounces), and the fluidity was controlled at 145° on the torsion viscometer, as previously described in slip preparation. The glaze was flocculated (to prevent run-marks on the finished piece) using a 50% solution of calcium chloride, giving the dipped and unfired piece a glaze

PHOTO 4. *Mould seams and surfaces of clay piece are carefully water-brushed to remove imperfections.*

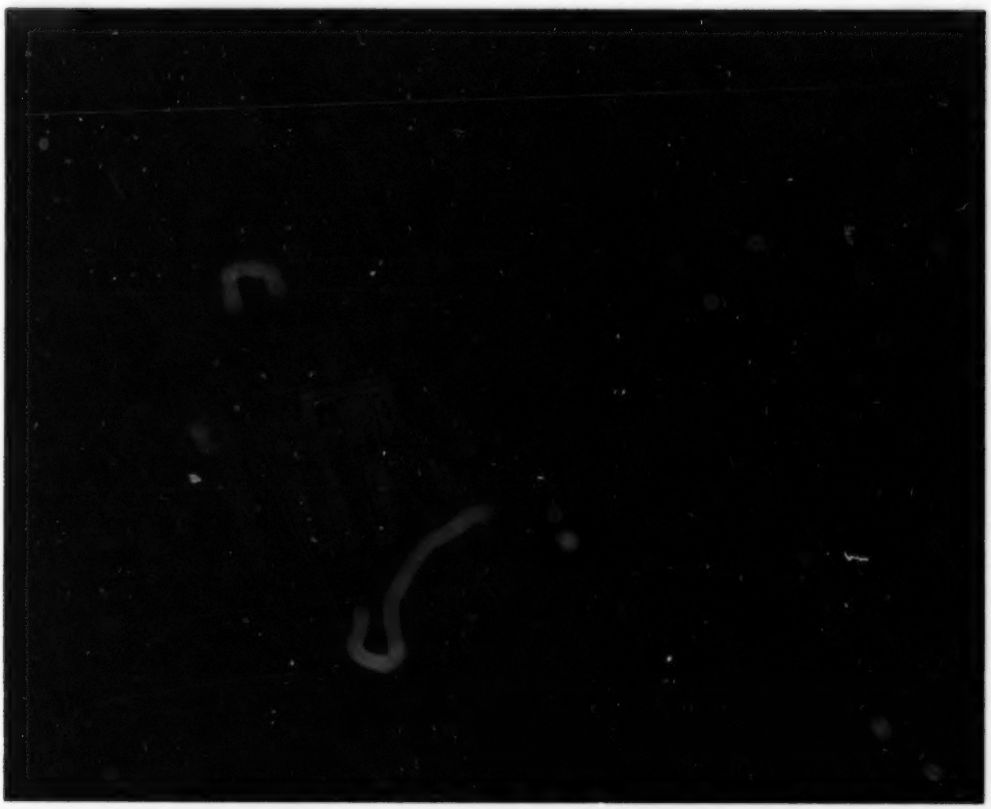


PHOTO 5. *Round clay piece placed on heat-resistant ring to prevent distortion during the first ('biscuit') firing.*

PHOTO 6. *Immersion of biscuit china in glaze. The item after coating with glaze is ready for firing at 1080° C.*

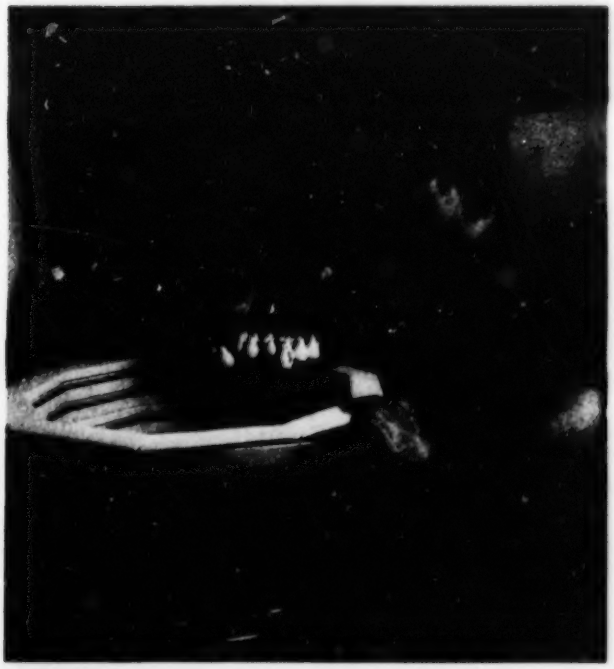


PHOTO 7. *Glazed pieces are placed in a high-temperature kiln. Pieces are kept separate to prevent fusion of adjacent items.*

thickness of 0.005 inches on the cups, and 0.0065 inches on the larger bowl. For thickness measurements, a glaze penetrometer was used.

The original 46% Ontario phosphate body was dipped in a white opaque glaze, so that the finished appearance was similar to bone china. This would need decorating with lithographs and gold to be a competitive product, and would not command as high a price as a translucent china body. Glaze control was applied exactly as with the translucent body, with specific gravity at 1.725 and fluidity at 145° on the viscometer.

All glazes were fired (Photo 7) to 1080°C and were found to be completely satisfactory. Figure 2 shows the firing curve.

DECORATING

Some pieces were left without decoration to allow examination of the body. Most cup-type items were decorated with lithographs and some with lithograph and 24-karat gold line at the top or extreme edges.

One small animal figure was hand-painted to indicate that such items could be produced of similar quality to the current fine china from England. Two miniature houses (pomanders) were also hand-painted for such comparisons.

The 8.5 inch presentation bowl was left, as far as the main body of the piece was concerned, without decoration. The surface modelling shows off the piece, indicating the potential for reproduction of hand-crafted china in the style of international giftware and high-priced tableware. A simple rose motif was applied to the interior base so as to set off the simplicity of the body and glaze appeal (Photo 8).

All decorative firing followed normal European bone china practice, in which the decorated piece is fired to a maximum temperature of 780°C (Figure 3), allowing any lithograph patterns to sink into the glaze with a consequent brilliance of finish and rendering the colours dish-washer resistant for domestic use. In all cases, the kiln dampers were left open up to the 400° mark, to allow any fumes from any fluxing or other additives to lithographs or gold to escape, thus ensuring a clean atmosphere in the kiln. The kiln was held at peak temperature for 30 minutes.

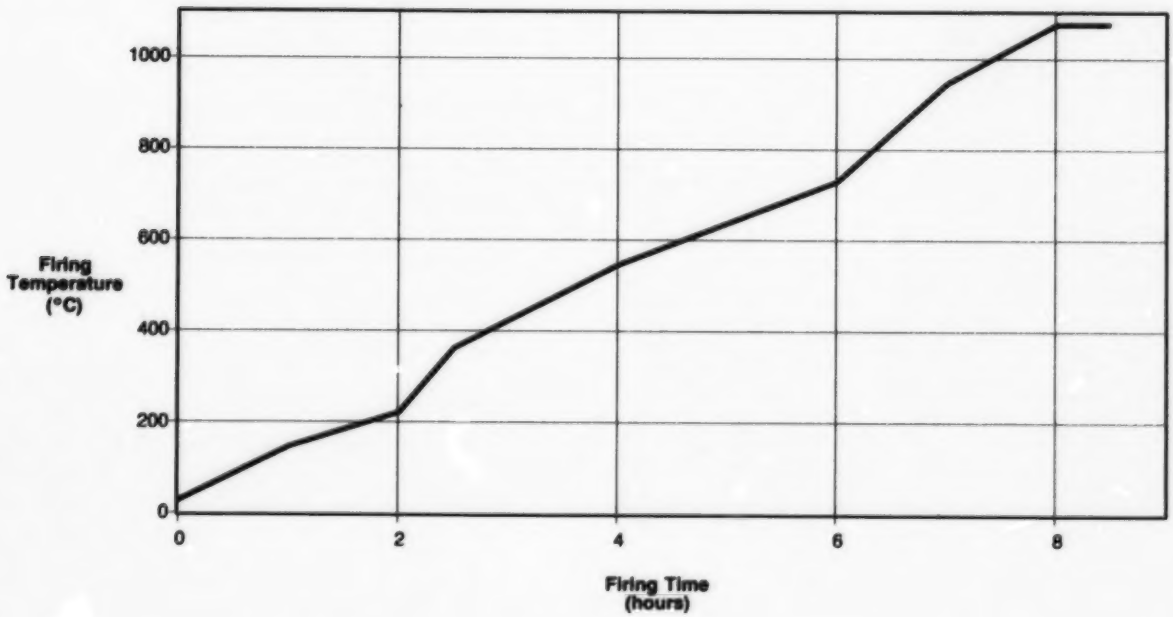


FIGURE 2. GLOST (GLAZED WARE) FIRING CURVE.

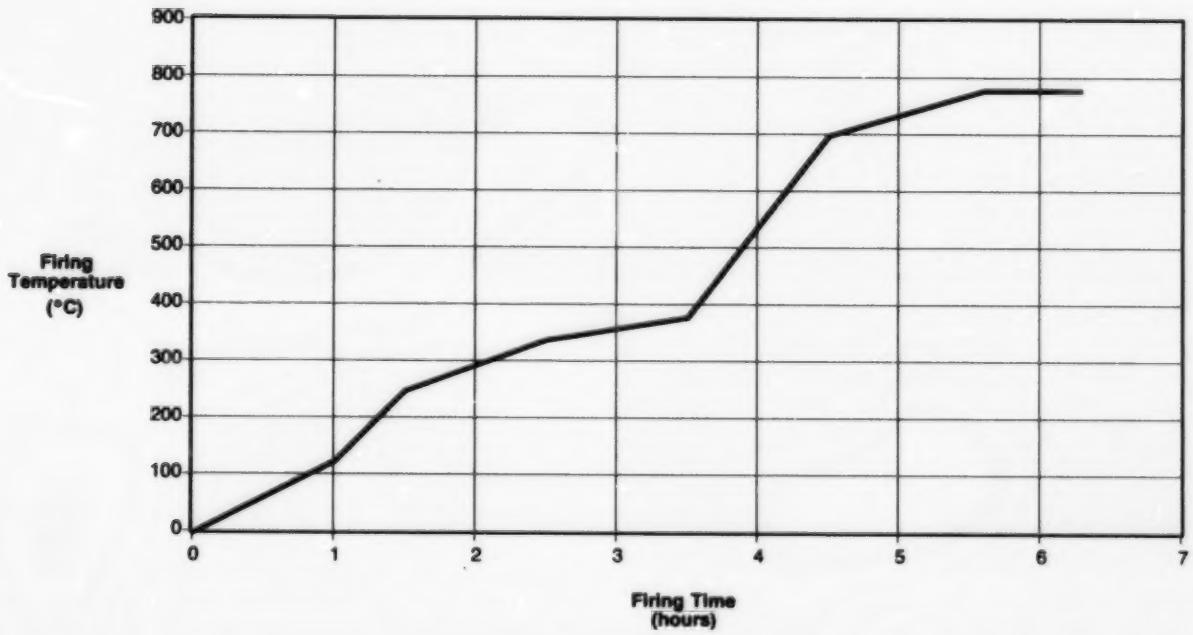


FIGURE 3. ENAMEL FIRING CURVE.

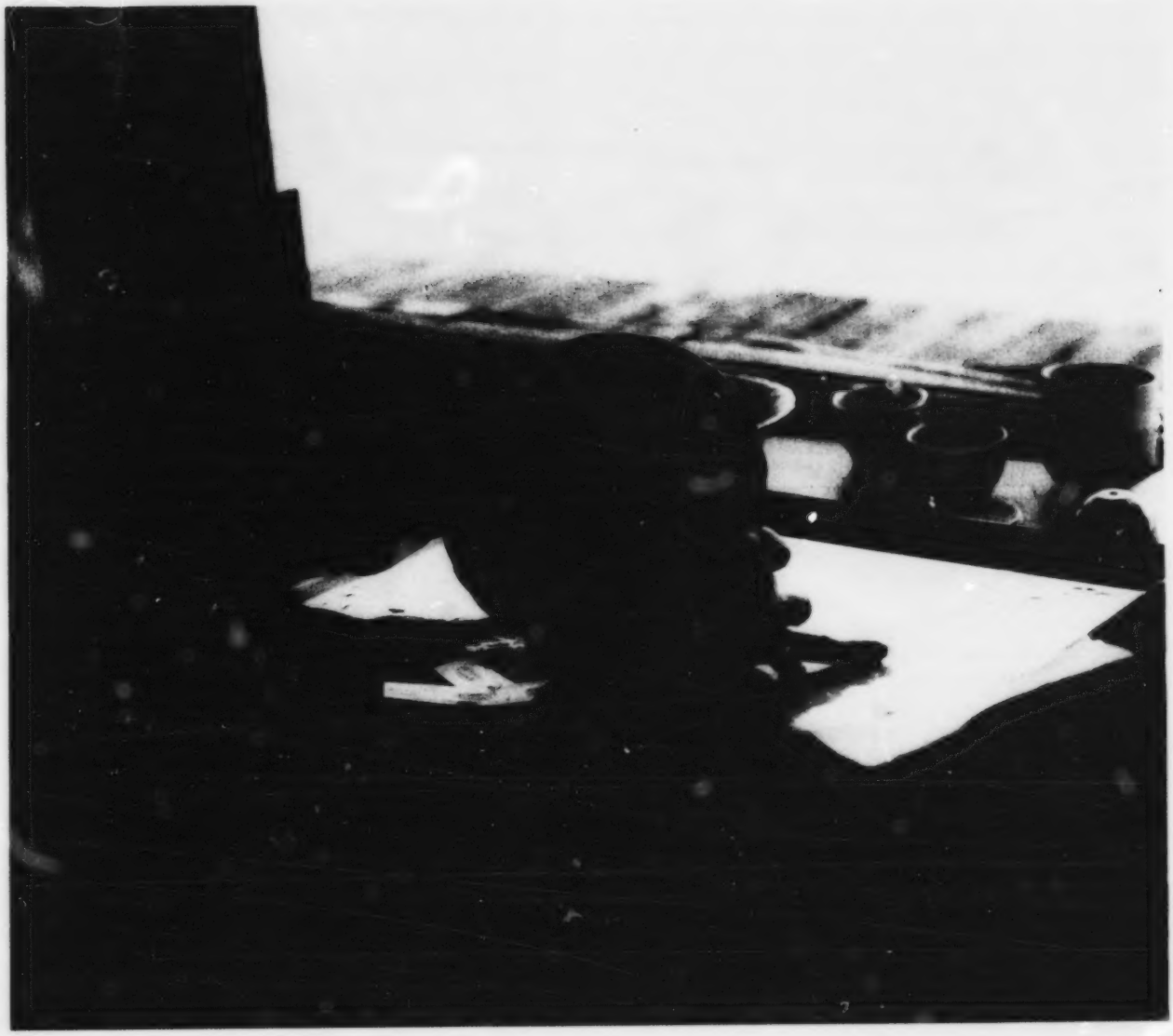


PHOTO 8. *Application of a lithograph to the interior base of a presentation bowl.*

5. Body Trials

The investigation of the use of Ontario materials was carried out by incremental replacement of standard English bone china materials. All bodies made in the trials were compared qualitatively to a classic bone china sample of English manufacture (the "Goss" body), which was denoted as **Body 1.**

The study was undertaken in the following stages:

Body 2/Body 3 Series — total replacement of bone ash constituent by Ontario rock phosphate; optimization of phosphate content, firing range and temperature.

Body 4 Series — additional substitution for Cornish stone by a blend of Ontario nepheline syenite and silica.

Body 5 Series — replacement of English china clay by Ontario kaolin; Ontario raw materials accounted for 100% of the composition of the products in this final series.

The progression of body development is charted in Figure 6 on page 25. Details of body composition and commentary on each stage of the manufacturing process are chronicled on pages 1 and 2.

BODY 2 SERIES

As stated earlier, typical fine bone china contains approximately 50% bone ash. The first phase of this study was to replace the bone with 46% Ontario phosphate (Batch 1 material).

The first bodies were fired at 1245°C for 6.5 hours, and a 1/2 hour soak (*see* Figure 4). The results were disappointing; the cups were badly blistered on the inside of the rims (Photo 9). Possible causes of the blistering were attributed as follows:

1. length of 'soak' at the elevated (1245°C) temperature;
2. dampness of the clayware before firing;
3. rapid rate of temperature increase in the early stages of firing;
4. excessive temperatures.

An encouraging note, however, was that the bodies retained their shape fairly well.

A second firing was carried out at a lower temperature of 1225°C. The firing time was increased from 6.5 to 9 hours with a 1-hour soak (Figure 4) and the pieces were completely dry before being placed in the ovens. No blistering occurred in the second firing.

A final, third firing was carried out at a lower temperature of 1215°C, with a slower rate of temperature rise, a longer firing time of 10 to 11 hours and with a 1/2 hour soak. This produced a sound vitreous body.

In all three firing trials, the pieces exhibited a brown-buff colour. This fell short of the whiteness associated with bone china. Cobalt chloride was added in varying amounts to several pieces in the third firing in an attempt to whiten the body; increases from 0.0125% to 0.025% lightened the colour moderately, but increasing the percentage further (maximum 0.037%) did little to improve the colour.

The bodies containing 46% phosphate failed to achieve any degree of translucency. Two causes for this were suspected: the phosphate material was not fine enough and/or the phosphate content was too high.

BODY 3 SERIES

The next phase of the trials was carried out by lowering the rock phospate content down to 12.2% (Batch 1 material). The resultant body (Body 3) was ivory in colour, and had a translucency comparable to internationally-renowned ivory china products. The best firing temperature, 1180°C (Figure 5, second firing), was 35°C less than that for the 46% phosphate body (*see* Figure 4). This firing curve was adopted for subsequent trials.

Subsequent tests at the 12.2% phosphate level (Body 3A) used the more finely-ground Batch 2 phosphate material. Virtually no difference, however, in colour or translucency was noted between the fired bodies 3 and 3A.

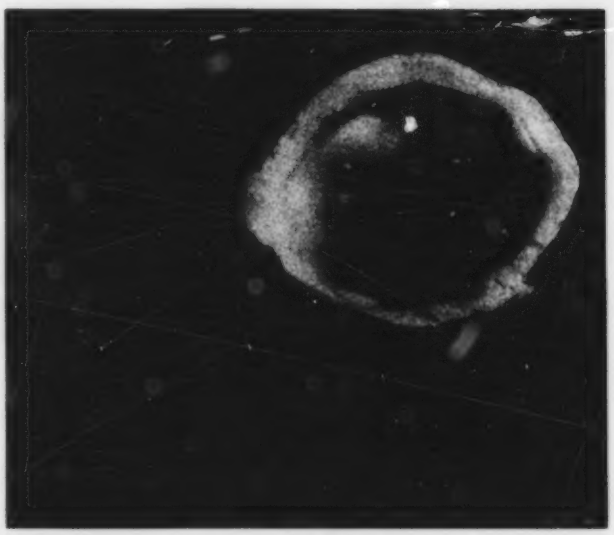


PHOTO 9. *Result of first firing at 1250°C of body with 46% Ontario phosphate content, showing severe blistering. Blistering was eradicated by firing at 1215°C.*

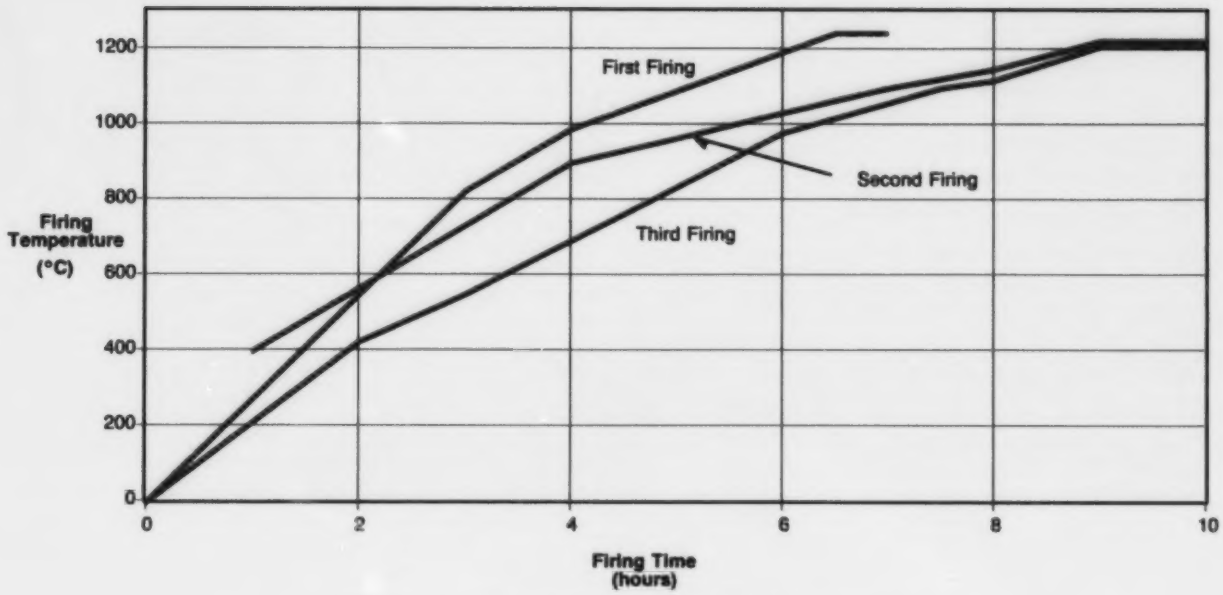


FIGURE 4. FIRING CURVE FOR BODY 2 SERIES (46% ONTARIO PHOSPHATE CONTENT).

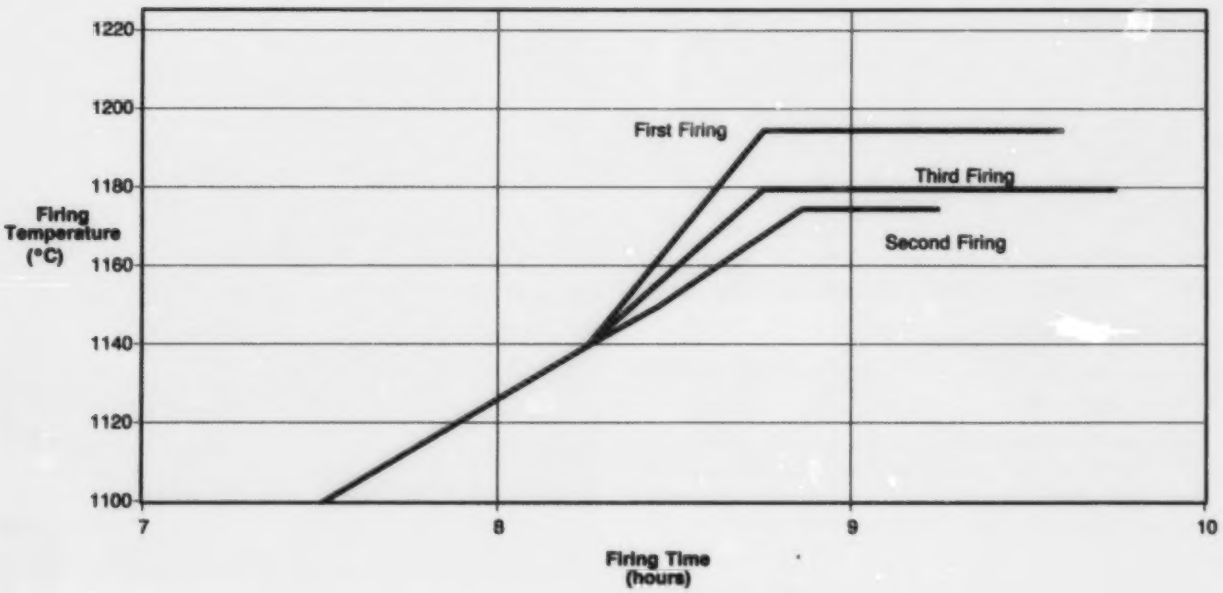


FIGURE 5. FIRING CURVE FOR BODY 3 SERIES — 3 AND 3A (12.2% ONTARIO PHOSPHATE CONTENT).

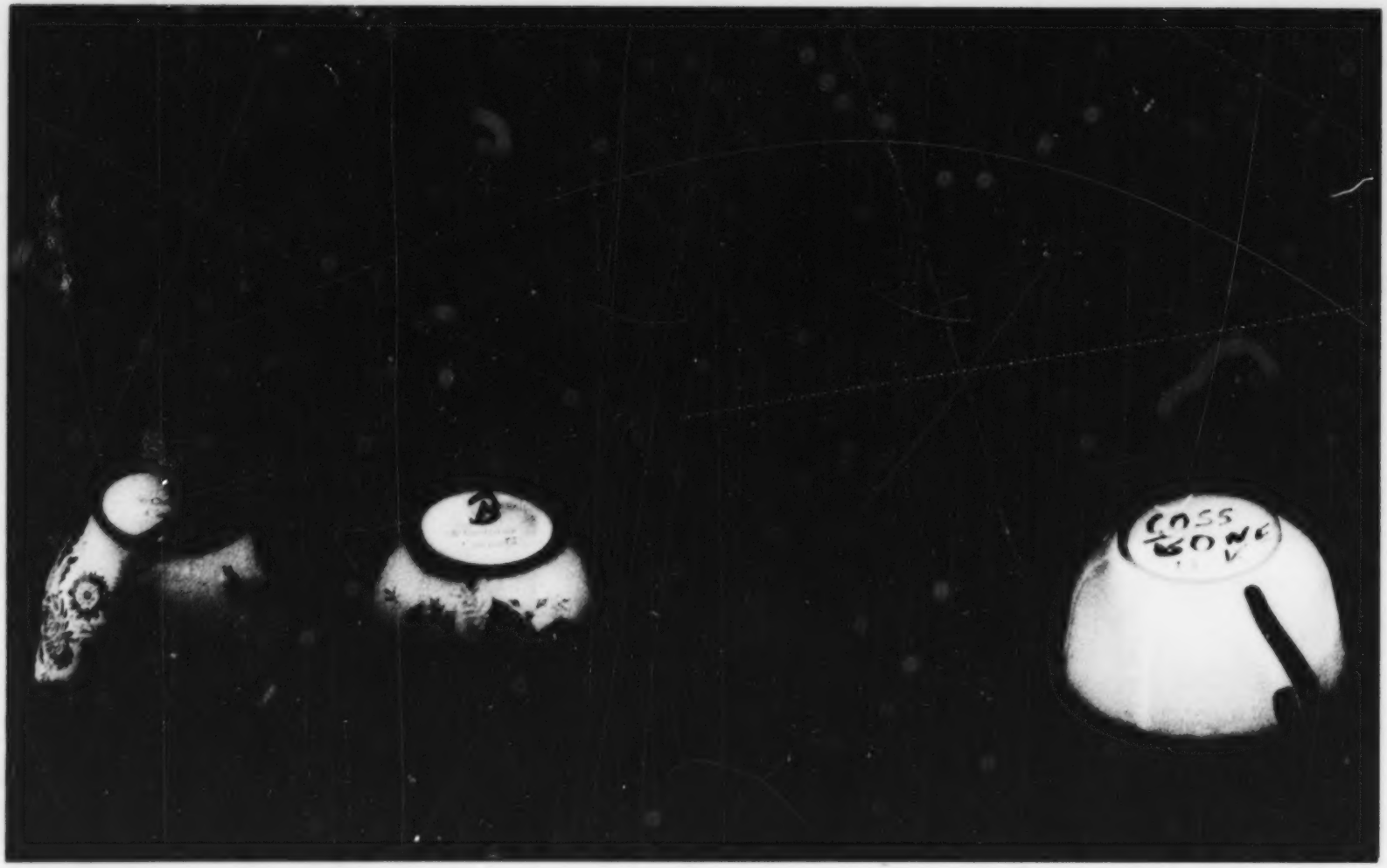


PHOTO 10. *Comparative translucencies of standard bone china and pieces containing Ontario phosphate. From left to right: Body 3A with 12.2% phosphate; Body 3B with 17.2% phosphate; Body 3C with 21.7% phosphate; bone china standard ("Goss" product). Note the increase in translucency with the decrease in rock phosphate content.*

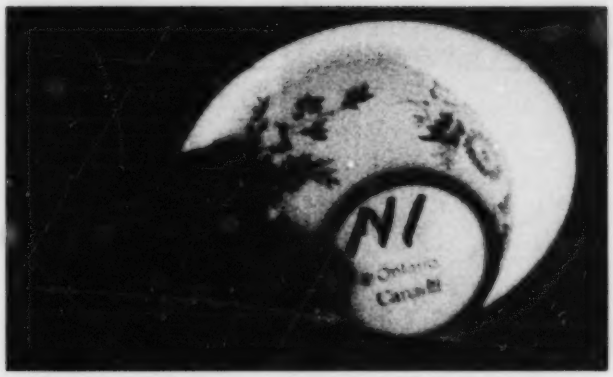


PHOTO 11. *Body 4–N1, showing good translucency. Body contains 66% combined Ontario phosphate, nepheline syenite and silica and 34% imported china clay. Level of cobalt chloride stain was at standard level of 0.0125%.*

PHOTO 12. *Body 4–N2 (or 4–NN), with the same composition as Body 4–N1 in Photo 11, but with twice the amount of cobalt chloride stain. No variation in translucency and no significant difference in whiteness was noted between the two bodies.*

The percentage of phosphate (Batch 2 material) was increased to 17.2% (Body 3B) and then 21.72% (Body 3C). Each increase, however resulted in a less translucent body (*see* Photo 10). The body with 21.72% phosphate content, however, was still comparable to fine porcelain available on the international market.

It was concluded from this phase of the trials that the phosphate content directly influenced the translucency. The finer particle sizing of the phosphate material, on the other hand, did not seem to influence the degree of translucency.

BODY 4 SERIES

With an acceptable standard of translucency achieved at the 12.2% phosphate level, English Cornish stone was replaced with nepheline syenite and silica in the next stage of the trials. The chemical composition of the Cornish stone was compared with nepheline syenite and silica to establish an equivalent blend.

Nepheline syenite is relatively high in alumina and total fluxes (CaO, Na_2O, K_2O) and low in silica. The reduction in fluxes to match those in Cornish stone resulted in a more closely–matched flux and alumina content. To balance the difference in SiO_2, silica was added in the proportions of 39% silica and 61% nepheline syenite. If the china body originally contained 53.7% Cornish stone, the final nepheline syenite content in the translucent body was calculated to be 32.7%; similarily, the silica content was 20.9%. Table 3 illustrates the chemical composition of the combined nepheline syenite and silica material in comparison with that of the Cornish stone standard.

Body 4 containing a total of 65.9% Ontario material maintained a good degree of translucency, similar to Body 3A (12.2% phosphate), and was ivory white in colour. The substitution of Ontario silica and nepheline syenite for Cornish stone

TABLE 3. CORNISH STONE AND NEPHELINE SYENITE/SILICA BLEND: EQUIVALENT CHEMICAL COMPOSITION.

	Cornish Stone	61%/39% Nepheline Syenite/Silica
SiO_2	72.38	75.20
Al_2O_3	14.85	14.15
Fe_2O_3	0.29	0.05
MgO	0.11	0.03
CaO	1.77	0.36
Na_2O	3.29	6.28
K_2O	4.60	2.87
TiO_2	0.02	–
LOI	1.87	–
TOTAL	99.18	98.94

was a complete success (Photos 11 and 12), with no loss of quality.

BODY 5 SERIES

In the final trials, Body 5 was made exclusively of Ontario materials. The composition was the same as Body 4, with the exception that English china clay was replaced in exact proportions by Ontario kaolin. There was a slightly discernable diminution in translucency as a result of the substitution of provincial kaolin; this was attributed to the level of iron content, and further refinement of the clay would be expected to eliminate this minor effect. The final product was a translucent, ivory-coloured body suitable for high-quality tableware (Photo 13) and giftware (Photos 14 and 15).

PHOTO 13. *Body 5, showing 8.5 inch diameter presentation bowl of ivory-coloured translucent fine china. The piece is composed entirely of Ontario materials with 12.2% phosphate content and with a transparent glaze.*

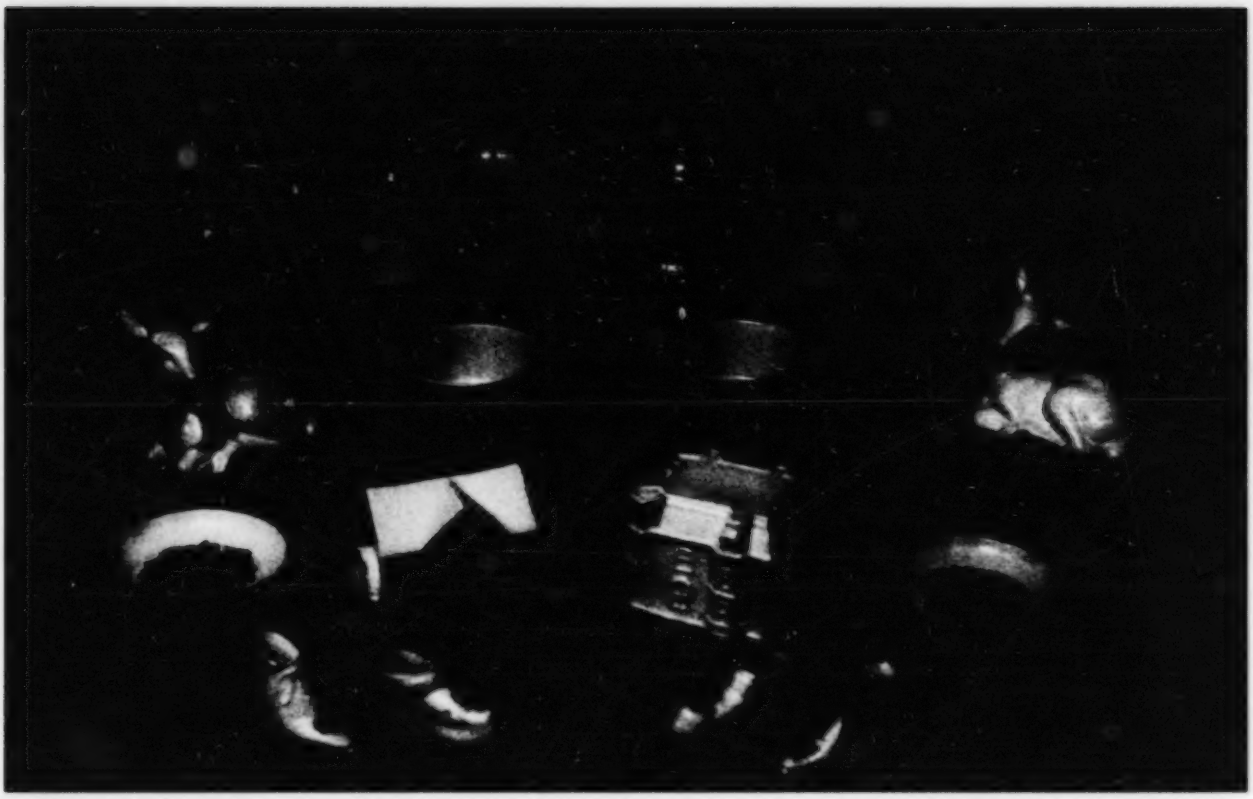


PHOTO 14. *Ensemble of products with opaque or transparent glazes, representing Body Series 3, 4 and 5. This selection shows the enhancement of the pieces by decoration with lithographs or by surface modelling.* **Rear:** *Cups, saucers and bell with Ontario mineral content of 12 to 22% — Body 3 series;* **Middle:** *Two bowls with Ontario mineral content of 66% — Body 4 series;* **Middle and Front:** *Giftware of figurines (deer, miniature houses and shoes) and plates with Ontario mineral content at 100% — Body 5 series.*

PHOTO 15. *Hand-painted opaque figurines produced in the Body 5 series (100% Ontario mineral content) show similar quality to imported china pomanders and animal figurines.*

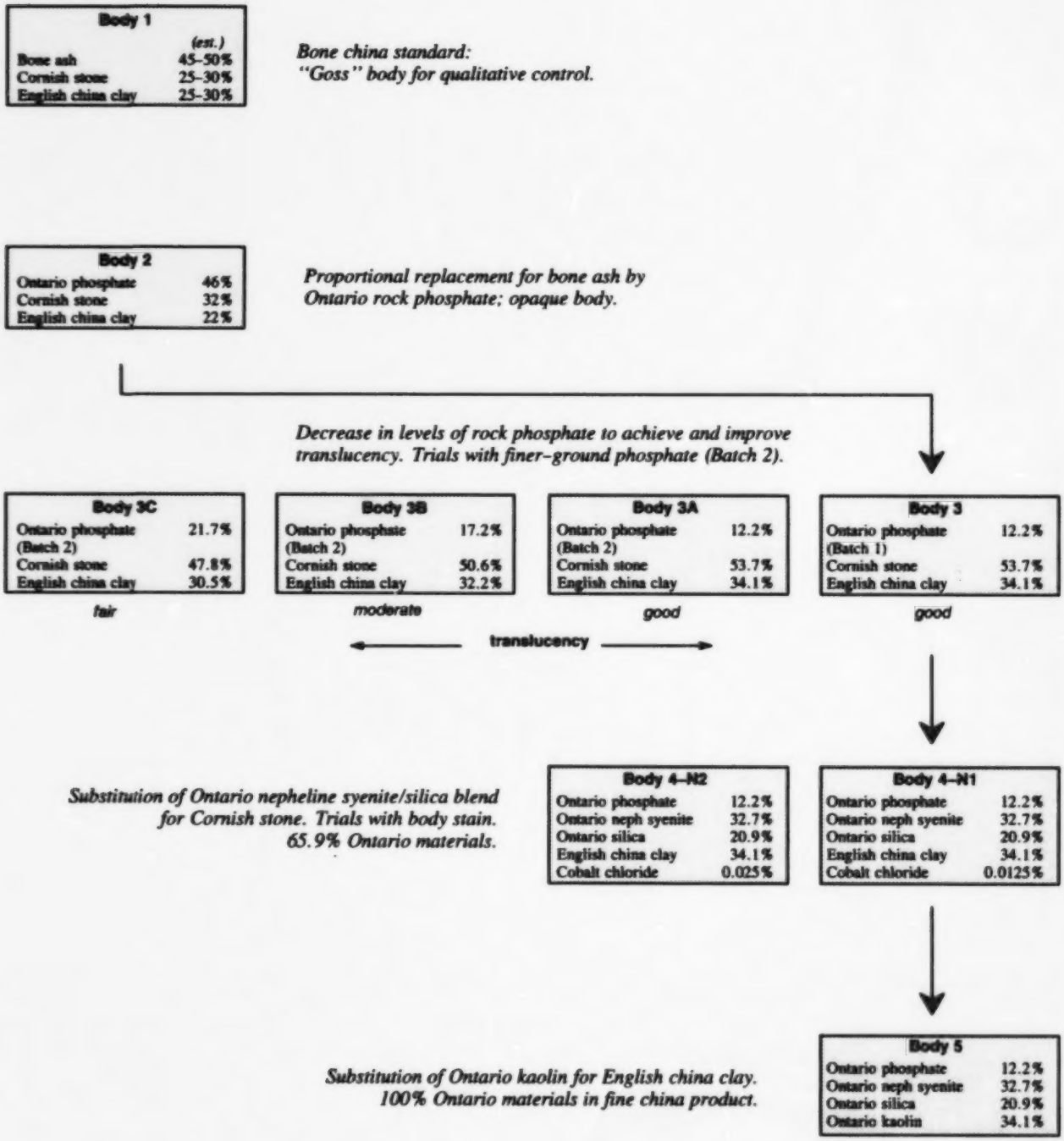


FIGURE 6. BODY DEVELOPMENT FLOW CHART.

PROCESSING COMPARISONS AND RESULTS

Process/Characteristic	Body 2 Series			Body 3 Series				Body 4 Series		Body 5 Series
1. Body composition (%)	**2A**	**2B**	**2C**	**3**	**3A**	**3B**	**3C**	**4-N1**	**4-N2**	
Ontario phosphate	46			12.2	12.2	17.2	21.7	12.2	12.2	12.2
Ontario nepheline syenite	–				–	–	–	32.7	32.7	32.7
Ontario silica	–				–	–	–	20.9	20.9	20.9
Ontario kaolin	–				–	–	–	–	–	34.1
English china clay	32			34.1	34.1	32.3	30.4	34.1	34.1	–
English Cornish stone	22			53.7	53.7	50.6	47.8	–	–	–
Cobalt chloride	0.0125	0.025	0.0375	0.0125	0.0125	0.0125	0.0125	0.0125	0.025	0.0125
Phosphate particle size:										
< 10 μm (Batch 1)	80	80	95	95	–	–	–	95	95	95
< 5 μm (Batch 2)	–	–	–	–	83	83	83	–	–	–
2. Body mixing (2 hr, high speed)	No problems.			No problems.				No problems.		Some problem with Ontario kaolin 'balling up'.
3. Sieving & magnetic treatment	Very little residue on sieves or magnets.			Very little residue on 150's sieve or magnet.				Very little residue on 150's sieve or magnet.		Additional silica on 150's mesh from kaolin.
4. Deflocculation	0.08% sodium silicate of S.G. 1.375 (75° Twaddle), plus refining.									
5. Casting time	3–5 min from filling to tipping.									
6. Draining time	4–5 min Drained well; no marking.			4–5 min				4–5 min		4–5 min
7. Scrapping time (from tipping time)	4–5 min No pulling, tearing or cracking.			4–5 min No pulling or cracking.				4–5 min		4–5 min No pulling or cracking.
8. Mould release time	45–60 min at room temperature. No distortion. Firm cast.			45–60 min at room temperature.				45–60 min at room temperature.		45–60 min at room temperature. Bowl allowed to rest until dry to prevent distortion.
9. Fettling & sponging	Must be dry or would get sticky. Must use minimum water.			Sponged well (no stickiness even with excess water).				No sticking. Took water well.		No sticking: could take extra water.
10. Placing	Round pieces on refractory rings, with alumina powder between.			Round pieces on refractory rings, with alumina powder between.				Round pieces on refractory rings, with alumina powder between.		Round pieces on refractory rings; Bowl (free standing) fired without distortion.

Process/Characteristic	Body 2 Series	Body 3 Series	Body 4 Series	Body 5 Series
11. Biscuit firing				
First	1245°C, 7 hr firing. 1/2 hr soak. Severe blistering on inside of cup. No translucency.	1195°C, 8.75 hr firing. 1 hr soak. Slight blistering. Signs of translucency.	1180°C, 9.5 hr firing. 1 hr soak. Good translucency. Body 4–N2 whiter. Firing curve now standard for bodies 4 & 5.	1180°C, 9.5 hr firing. 1 hr soak. Good translucency. Slight diminution as compared to body 4. Good ivory colour.
Second	1225°C, 9 hr firing. 1 hr soak. No blistering or distortion. No translucency.	1175°C, 8.75 hr firing. 1/2 hr soak Slight translucency.		
Third	1215°C, 10.5 hr firing. 1/2 hr soak. No blistering or distortion. No translucency. Light brown, non-porous.	1180°C, 8.75 hr firing. 1 hr soak. Good translucency. No blistering or distortion. No difference in translucencies between 3 and 3A bodies in firings.		
12. Contraction	12%	10.4%	9.6%	11.2%
13. Water absorption				
Firing 1	Not determined.	Not determined.	Not determined.	Not determined.
Firing 2	0.508%–1.210%			
Firing 3	0.32%			
14. Glazing	..... Fluidity 145 on torsion viscometer, using 30's gauge wires and 11/16" cylinder.			
	..... S.G. of glaze, 1.725, unfired thickness is 0.005".			
	a) Transparent, low-solubility: brown china. b) White opaque: white.	a) Transparent, low-solubility china type.	a) Transparent, low-solubility china type.	Used CaCl for flocculation. a) Transparent, low-solubility china type.
15. Glost firing	1080°C, 1/2 hr soak.	1080°C, 1/2 hr soak.	1080°C, 1/2 hr soak.	1080°C, 1/2 hr soak.
16. Decorating	On-glaze lithographs. 24 karat gold trim Firing at 780°C. 1/2 hr soak. Good, bright finish.	Lithograph & gold. Fired at 780°C. 1/2 hr soak. Good finish. No cracking of gold.	Lithograph & gold. Fired at 780°C. 1/2 hr soak. Good colours, no gold cracking.	Lithograph. Figurine painted to show versatility of decorating possibilities. Good colours; no gold used.

6. Results and Conclusions

The preliminary investigation of the suitability of selected Ontario minerals to replace the traditional components of a bone china body resulted in the trial production of an attractive translucent fine china. The principal results were as follows:

1. The trial substitution of calcined bone by beneficiated Ontario phosphate from the Cargill Township deposit did not produce a body of the translucency and whiteness associated with bone china of premier quality.

   The use of rock phosphate at various levels failed to produce a white body. No improvement was obtained by the application of cobalt chloride stain at up to three times the standard level of usage. This was principally attributed to the iron and titiania content of the phosphate material used in the trials.

   The replacement of bone ash in equal proportion by phosphate produced an opaque body. Comparable translucency to commercial fine china grades, however, was generally achievable at levels below 20% rock phosphate content; the degree of translucency increased with corresponding decreases in phosphate content. No improvement in translucency was observed in the use of finer particle sizing (minus 5 microns) in comparison with the sizing (minus 10 microns) used for the phosphate material in the majority of the test bodies.

2. Commercially available nepheline syenite and silica ceramic-grade products, derived from deposits in Peterborough County and Badgeley Island (Georgian Bay) respectively, were shown to be successful replacements for Cornish stone in chemically equivalent proportions and without further treatment.

3. Treated kaolin from the Mattagami River basin deposit was an adequate replacement for English china clay, confirming prior ceramic work with this material.

4. The substitution of Ontario nepheline syenite, silica and kaolin was undertaken with the rock phosphate content held at 12%. Good translucency was maintained throughout the additional stages of material substitution. Nevertheless, both potential kaolin and phosphate products from Ontario sources would appear to require substantial upgrading with respect to purity and consistency to fully meet commercial standards for the manufacture of fine china tableware.

5. Rock phosphate content at the 46% level produced a pale brown, opaque but non-distorted body. This was judged to be a sound body which, with opaque glazing, would be suitable for regular hotel and restaurant tableware and lend itself to the manufacture of varied giftware, although lacking in the aesthetic appeal and price potential of a translucent body.

   It was also considered that lithographic or other decoration would be required to provide market potential for a manufactured product.

6. The trials established that a formulation of 12% phosphate, 34% kaolin, 33% nepheline syenite and 21% silica can produce a vitreous china of good translucency and attractive ivory colour.

   The body will 'pot' at minimal thicknesses, giving it a fine, handcrafted look, and fires well. Using a transparent glaze and standard cobalt chloride stain, this product is judged to be particularly appropriate for tableware and giftware of high quality. It is eminently suitable for the use of surface relief with sculptural patterns and embossed motifs.

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